

Filled expandable styrenic particles**Publication number:** EP1002829**Publication date:** 2000-05-24**Inventor:** GLUECK GUISCARD DR (DE); HAHN KLAUS DR (DE);
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C08K3/00P5; C08K7/00**Application number:** EP19990121808 19991104**Priority number(s):** DE19981052683 19981116**Also published as:**

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Report a data error here**Abstract of EP1002829**

Particulate expanded styrene polymer with good liquid uptake contains a solid that is insoluble in water and a hydrocarbon. Particulate expanded styrene polymer (EPS particles) contain 1-25 wt. % of a solid that is insoluble in water and hydrocarbons, has an average particle size of above 50 μ m and, when in needle form, a length of over 20 μ m. Independent claims are included for the production of open-celled polystyrene particulate foams by foaming and sintering the above polymer and the obtained foams.

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Open-cell molded polystyrene foams

The invention relates to expandable polystyrene beads (EPS beads), and also to open-cell molded polystyrene foams which can be produced therefrom by foaming.

Compared with close-cell foams, open-cell foams have better sound-insulation properties and increased liquid absorption. This can be advantageous in some applications. Open-cell molded polystyrene foams have not yet been disclosed. However, there is a need for foams of this type since it is likely that they will have other advantageous properties, e.g. good heat-insulation properties.

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Patent Application WO 96/34039 describes open-cell microcellular polystyrene foams with an average cell size of 70 μm or below. These are not molded foams but foams produced directly by foam extrusion. The examples describe a large number of foams, the lowest density being 29.12 g/l, the greatest cell size 68 μm , and the lowest open-cell content 84.1%. The foams are produced by extruding polystyrene and blowing agents in the presence of finely divided carbon black or graphite particles. The latter act as nucleating agents and are responsible for the low cell size. The open-cell nature of the foams derives from bursting of the small cells when the polymer melt passes through the extrusion die. The open-cell microcellular foams of WO 96/34 039 are claimed to have low thermal conductivity, in particular when they have been evacuated, but no other advantageous properties have been described.

Patent Applications PCT/EP 97/02457 and PCT/EP 97/02458 propose incorporating graphite particles in expandable polystyrene beads. This can reduce the thermal conductivity of foams prepared therefrom. The graphite particles used here have a particle size of preferably from 1 to 50 μm . However, when EPS beads of this type are foamed and sintered the result is not an open-cell foam with an open-cell content of more than 5%.

The invention provides EPS beads which comprise from 1 to 25% by weight, preferably from 2 to 20% by weight and in particular from 3 to 10% by weight, of a solid which is insoluble in water and in hydrocarbons having from 4 to 10 carbon atoms and has an average particle size above 50 nm or, in the case of acicular shapes, a length above 20 μm .

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The solids are to be insoluble, i.e. they must not dissolve or begin swelling in water, styrene or pentane during the preferred preparation of the EPS beads by polymerizing styrene in aqueous suspension in the presence of pentane. The average particle size is determined by Fraunhofer diffraction, particle size being the longest particle diameter. The average particle size is to be above 50 μm , preferably from 60 to 200 μm and in particular from 80 to 160 μm . In the case of acicular solids, i.e. those whose length-to-diameter ratio is above 5:1, the length is to be more than 20 μm , preferably more than 40 μm , in particular more than 50 μm .

Preferred solids are graphite particles and hydrophobicized glass fibers, and also hydrophobicized silicates, metal pigments and metal oxides, and also polymer resins, preferably composed of crosslinked polymers.

The novel EPS beads comprise, as polymer matrix, in particular homopolystyrene or styrene copolymers with up to 20% by weight, based on the weight of the polymers, of ethylenically unsaturated comonomers, in particular alkylstyrenes, divinylbenzene, acrylonitrile or α -methylstyrene.

The EPS beads are preferably prepared by polymerizing styrene, if desired together with 20% of its weight of comonomers, in aqueous suspension in the presence of the solid and with addition of blowing agents prior to, during or after the polymerization.

The usual auxiliaries may be used in this suspension polymerization, for example peroxide initiators, suspension stabilizers, blowing agents, chain transfer agents, expansion aids, nucleating agents and plasticizers. Particular preference is given to the addition of flame retardants, preferably in amounts of from 0.1 to 6% by weight, and of flame retardancy synergists in amounts of from 0.1 to 1% by weight, based in each case on the resultant styrene polymer. Preferred flame retardants are aliphatic, cycloaliphatic and aromatic bromine compounds, such as hexabromocyclododecane, pentabromomonochlorocyclohexane and pentabromophenyl allyl ether. Suitable synergists are C-C- or O-O-labile organic compounds, such as dicumyl and dicumyl peroxide. The amounts of blowing agents added are from 3 to 10% by weight, based on polystyrene. They may be added prior to, during or after the polymerization of the suspension. Suitable blowing agents are aliphatic hydrocarbons having from 4 to 6 carbon atoms. It is advantageous to use Pickering dispersing

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agents as suspension stabilizers, for example magnesium pyrophosphate or calcium phosphate.

The solid particles added during the course of the suspension
5 polymerization are usefully suspended in advance in an organic solvent, preferably in styrene. It is particularly useful to use a solution of polystyrene in styrene as suspension medium. It is also possible to feed graphite in the form of a polystyrene masterbatch. The weight ratio of solid to suspension medium here
10 is preferably from 80 : 20 to 20 : 80, in particular about 50 : 50.

The suspension of the solid is added to the polymerization mixture at a monomer conversion of from 10 to 100%, preferably
15 from 20 to 90% and in particular from 25 to 75%.

In principle the novel EPS beads may also be prepared by incorporating the solid particles into a melt of polystyrene and blowing agent in an extruder and pelletizing the extrudate. No
20 foaming takes place here.

The expandable polystyrenes comprising solid particles may be processed to give open-cell polystyrene foams.

25 For this, the expandable particles are prefoamed. This is mostly done by heating the particles with steam in what are known as prefoamers. The particles prefoamed in this way are then fused to give moldings. For this, the prefoamed particles are introduced into non-gas-tight molds, and steam is applied. After cooling,
30 the moldings can be removed.

It is likely that when the EPS beads are foamed the cell membranes which form are broken up by the relatively large solid particles to give open cells.

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The invention also provides open-cell molded polystyrene foams with a density of from 5 to 50 g/l, preferably from 10 to 25 g/l, an average cell size of from 50 to 1000 μm , preferably from 75 to 750 μm , and in particular from 100 to 500 μm , and an open-cell
40 content of from 5 to 80%, preferably from 10 to 65% and in particular from 20 to below 70%.

The open-cell molded polystyrene foams, unlike the microcellular foams of WO 96/34 039, are generally used in non-evacuated form.

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The cell size is measured by measuring and counting cell diameters on microtome sections of the foams. The open-cell content is determined to ASTM D 2856-87, method C, using an Accupyc 1330 apparatus.

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The novel foams have not only excellent heat insulation properties but also good sound-insulation properties and low shrinkage. The latter is important when producing relatively large moldings, e.g. for foam slabs, in which there is a tendency for depressions to form after removal from the molding machine and also for shrinkage to occur, even after some days. The open-cell nature of the foams means that they can absorb liquids and are therefore suitable as drainage sheets and can, for example, be used to absorb oil from tanker accidents.

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Example 1

61.0 g of dicumyl peroxide, 40.0 g of dicumyl, 15 g of hexabromocyclododecane and 20.2 g of dibenzoyl peroxide are dissolved in 18.4 kg of styrene. The organic phase is introduced into 20.0 l of deionized water in a 50 l mixing vessel. The aqueous phase comprises 35.0 g of sodium pyrophosphate and 70.0 g of magnesium sulfate (Epsom salt). The suspension is heated to 80°C. After 110 minutes, at a styrene conversion of about 30%, 1000 ml of styrene in which 650 g of graphite with an average particle size of 75 µm (KP 99.5; Graphitwerk Kropfmühl) have been suspended are metered in. After a further 30 minutes, 1.8 g of K 30 emulsifier (Bayer AG) are added. After a further 30 minutes, 1.60 kg of pentane are fed in and polymerization is completed at 125°C. The resultant polystyrene beads comprising blowing agent are isolated by decanting, washed and dried. They are foamed by a conventional method to give foam beads, which are sintered to give a foam slab of density 10 g/l. The average cell size is 300 µm and the open-cell content is about 20%.

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Example 2

61.0 g of dicumyl peroxide, 40.0 g of dicumyl, 15 g of hexabromocyclododecane and 20.2 g of dibenzoyl peroxide are dissolved in 18.4 kg of styrene. The organic phase is introduced into 20.0 l of deionized water in a 50 l mixing vessel. The aqueous phase comprises 35.0 g of sodium pyrophosphate and 70.0 g of magnesium sulfate (Epsom salt). The suspension is heated to 80°C. After 110 minutes, 1000 ml of styrene are metered in. The styrene comprises 650 g of silylated glass fibers (fiber diameter about 3 µm, fiber length about 100 mm), i.e. 3% based on the entire organic phase. After a further 30 minutes, 1.8 g of K 30

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emulsifer (Bayer AG) are added. After a further 30 minutes, 1.60 kg of pentane are fed in and the polymerization is completed at 130°C. The resultant EPS beads are again processed to give an open-cell foam.

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We claim:

1. An expandable polystyrene bead (EPS bead) which comprises
5 from 1 to 25% by weight of a solid which is insoluble in water and in hydrocarbons having from 4 to 10 carbon atoms, wherein the solid has an average particle size above 50 μm or, in the case of acicular shapes, a length above 20 μm .
- 10 2. An EPS bead as claimed in claim 1, wherein the solid is hydrophobicized silicates, hydrophobicized glass fibers, polymer resins, hydrophobic metallic pigments, hydrophobic metal oxides or graphite particles.
- 15 3. A process for preparing open-cell molded polystyrene foams by foaming and sintering the EPS beads as claimed in claim 1.
4. An open-cell molded polystyrene foam which has a density of
20 from 5 to 50 g/l, an average cell size of from 50 to 1000 μm and an open-cell content of from 5 to 80%.
5. An open-cell molded polystyrene foam as claimed in claim 1, which has not been evacuated.
- 25 6. An open-cell molded polystyrene foam as claimed in claim 1, which has a density of from 10 to 25 g/l.
7. An open-cell molded polystyrene foam as claimed in claim 1,
30 which has an average cell size of from 80 to 500 μm .
8. An open-cell molded polystyrene foam as claimed in claim 1, which has an open-cell content of from 20 to below 70%.

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Open-cell molded polystyrene foams

Abstract

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Expandable polystyrene beads comprise a solid with an average particle size above 50 μm or, in the case of acicular shapes, a length of above 20 μm . These can be used to produce molded polystyrene foams with an open-cell content of from ~~10~~ to 80%.

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